



Crystal structures of two (Z)-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamides

Aleksei Galushchinskiy,^a Pavel Slepukhin^b and Konstantin Obyednov^{a*}

^aUral Federal University, Mira 19 Ekaterinburg 620002, Russian Federation, and ^b22 Sofia Kovalevskaya str., Ekaterinburg, 620990, Russian Federation. *Correspondence e-mail: k.l.obyednov@urfu.ru

Received 1 November 2017

Accepted 7 November 2017

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; thiazolidine; thiazolidin-4-one; acetamide; hydrogen bonding.

CCDC references: 1582018; 1582019

Supporting information: this article has supporting information at journals.iucr.org/e

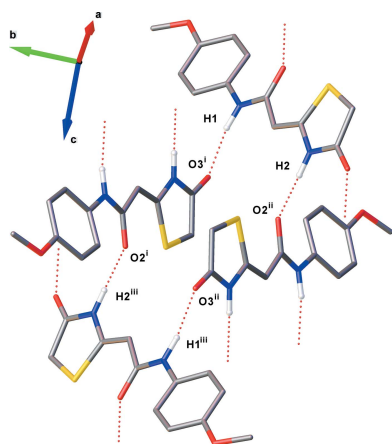
The crystal structures of two (oxothiazolidin-2-ylidene)acetamides, namely (Z)-2-[2-(morpholin-4-yl)-2-oxoethylidene]thiazolidin-4-one, C₉H₁₂N₂O₃S, (I), and (Z)-N-(4-methoxyphenyl)-2-(4-oxothiazolidin-2-ylidene)acetamide, C₁₂H₁₂N₂O₃S, (II), are described and compared with a related structure. The Z conformation was observed for both the compounds. In (I), the morpholin-4-yl ring has a chair conformation and its mean plane is inclined to the thiazolidine ring mean plane by 37.12 (12)°. In (II), the benzene ring is inclined to the mean plane of the thiazolidine ring by 20.34 (14)°. In the crystal of (I), molecules are linked by N—H...O hydrogen bonds, forming C(6) chains along the b-axis direction. The edge-to-edge arrangement of the molecules results in short C—H...O and C—H...S interactions, which consolidate the chain into a ribbon-like structure. In the crystal of (II), two N—H...O hydrogen bonds result in the formation of C(8) chains along the b-axis direction and C(6) chains along the c-axis direction. The combination of these interactions leads to the formation of layers parallel to the bc plane, enclosing R₄⁴(28) rings involving four molecules.

1. Chemical context

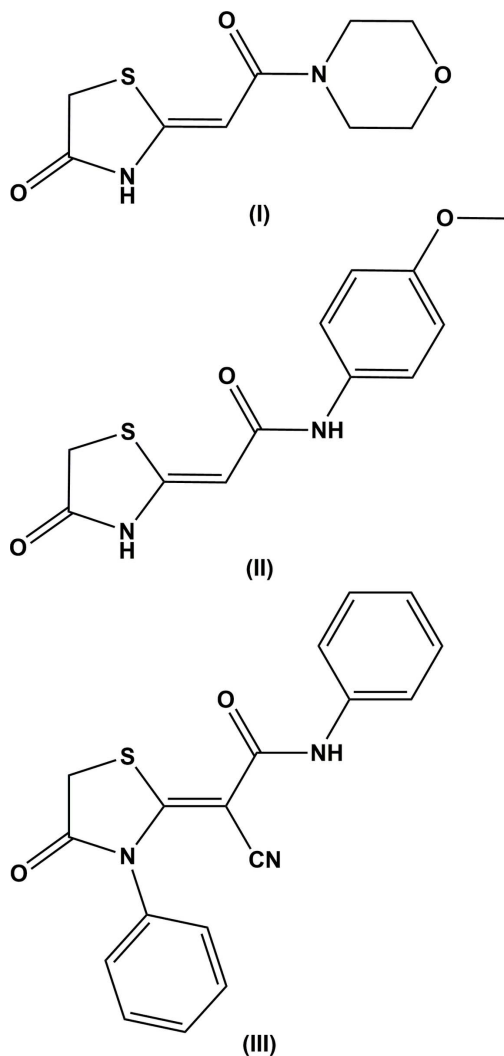
Thiazolidine derivatives are of great biological importance due to their antidiabetic (Rizos *et al.*, 2016) and antibacterial (Har & Solensky, 2017) activity. One such compound, namely (Z)-N-(2-chloro-6-methylphenyl)-2-(3-methyl-4-oxo-1,3-thiazolidin-2-ylidene)acetamide (ralitoline), has been found to be effective in a preclinical anticonvulsant evaluation (Löscher & Schmidt, 1994). In view of the importance of 2-(4-oxothiazolidin-2-ylidene)acetamides, the title compounds, (I) and (II), were synthesized and we report herein on their crystal structures. To date, the crystal structure of only one such compound, *viz.* (Z)-2-cyano-2-(4-oxo-3-phenyl-1,3-thiazolidin-2-ylidene)-N-phenylacetamide, (III), has been reported (George, 2012).

2. Structural commentary

The molecular structures of the title compounds, (I) and (II), are illustrated in Figs. 1 and 2, respectively. Both compounds crystallize in the monoclinic space group *P*2₁/*c*. The Z conformation about the C8=C9 bond is observed for both compounds and favours S...O contacts of 2.6902 (18) and 2.738 (3) Å in (I) and (II), respectively. The morpholine ring in compound (I) adopts a chair conformation. The twist angle between the thiazolidine (S1/N2/C9–C11) and amide mean planes (O1/N1/C7/C8) is 10.71 (10)° in (I) and 2.36 (14)° in (II). In (II), the benzene ring plane is inclined to the mean plane of the thiazolidine ring by 20.60 (12)°. The bond lengths



and angles in both compounds are similar to those observed for compound (III), mentioned above.



3. Supramolecular features

In the crystal of (I), molecules are linked by N—H...O hydrogen bonds forming *C*(6) chains running parallel to the

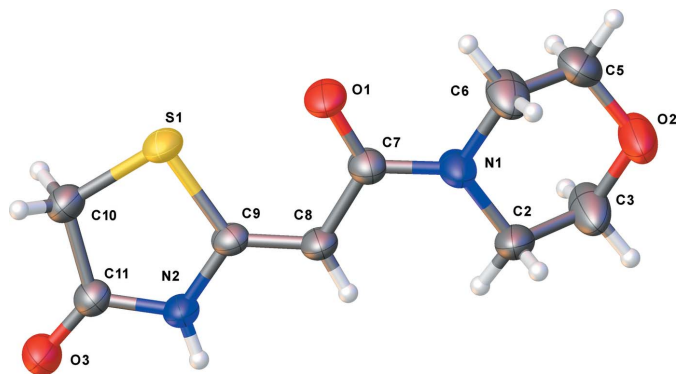


Figure 1
The molecular structure of title compound (I), with the atom labelling. Displacement ellipsoids at the 50% probability level.

Table 1
Hydrogen-bond geometry (Å, °) for (I).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2...O1 ⁱ	0.79 (3)	2.11 (3)	2.891 (2)	167 (2)
C2—H2A...S1 ⁱ	0.97	2.86	3.627 (2)	137
C5—H5B...O3 ⁱⁱ	0.97	2.55	3.503 (3)	167
C6—H6B...O3 ⁱⁱⁱ	0.97	2.41	3.179 (3)	136

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x + 1, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O3 ⁱ	0.95 (2)	1.94 (2)	2.883 (4)	170 (2)
N2—H2...O2 ⁱⁱ	0.93 (3)	1.92 (3)	2.828 (4)	164 (3)

Symmetry codes: (i) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

a-axis direction (Table 1 and Fig. 3). The dihedral angle between thiazolidine mean planes is 6.12 (7)°. There are three non-classical C2—H2A...S1ⁱ, C5—H5B...O3ⁱⁱ and C6—H6B...O3ⁱⁱⁱ (Table 1) hydrogen bonds present, linking molecules to form ribbons propagating along [100]; Table 1 and Fig. 3.

In crystal of (II), both amide moieties participate in the formation of N—H...O hydrogen bonds (see Table 2). These two types of N—H...O hydrogen bonds give rise to the formation of two independent *C*(8) and *C*(6) chains, running parallel to the *b*- and *c*-axes, respectively (see Figs. 4 and 5). Here, the dihedral angle between the thiazolidine mean planes in the N1—H1...O3ⁱ and N2—H2...O2ⁱⁱ motifs is 79.21 (16)°. The combination of these chain motifs generates a two-dimensional network lying parallel to the *bc* plane. Each molecule acts as both a double donor and a double acceptor of N—H...O hydrogen bonds. The molecules of (II) are linked into aggregated *R*₄⁴(28) tetramers, which serve as the building blocks of the layers (see Fig. 6).

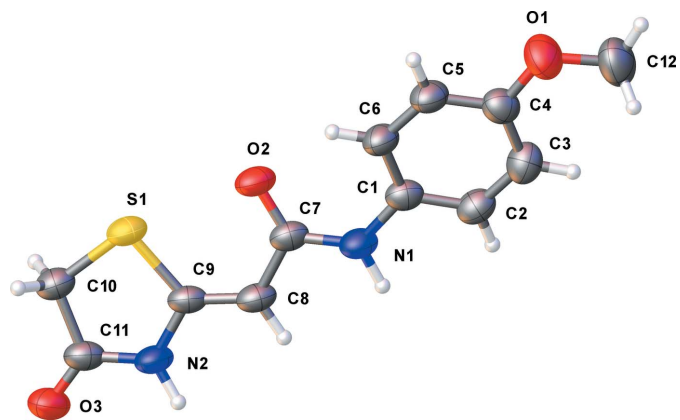


Figure 2
The molecular structure of title compound (II), with the atom labelling. Displacement ellipsoids at the 50% probability level.

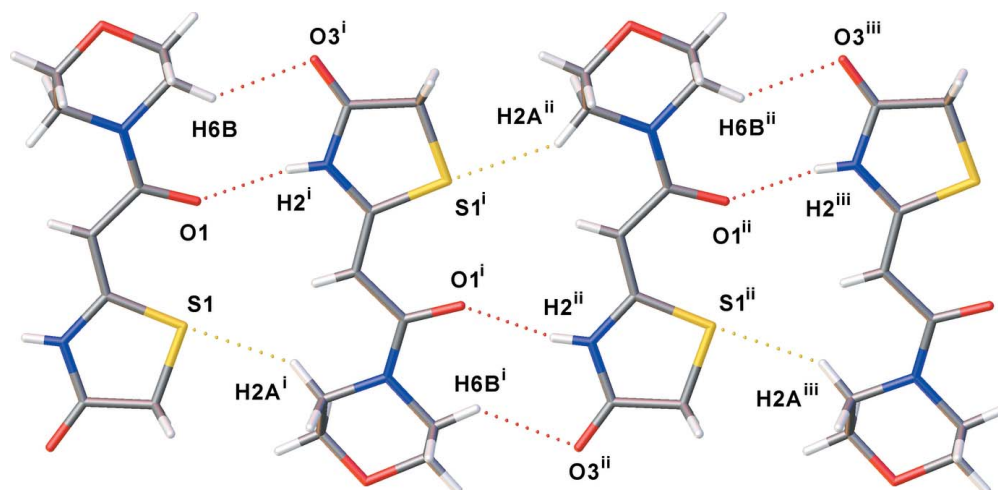


Figure 3
A packing diagram of compound (I). Dashed lines represent hydrogen bonds. [Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, y - 1, z$; (iii) $-x + 1, y - \frac{3}{2}, -z + \frac{3}{2}$.]

4. Database survey

A search of the Cambridge Structural Database (Version 5.38; Groom *et al.*, 2016) for the 2-methylene-1,3-thiazolidin-4-one substructure gave nine hits. The compound that most closely resembles the title compounds is 2-cyano-2-(4-oxo-3-phenyl-1,3-thiazolidin-2-ylidene)-*N*-phenylacetamide (III) (NEYGUV; George, 2012). Here the amide mean plane [C—C(=O)—N] is inclined to the mean plane of the thiazolidine ring by $5.09 (16)^\circ$, compared to $2.36 (14)^\circ$ in (II). The benzene ring is inclined to the mean plane of the thiazolidine ring by $38.10 (15)^\circ$ compared to $20.34 (14)^\circ$ in (II). In the crystal of (III), molecules are linked by N—H \cdots O hydrogen bonds, forming chains along the [010] direction. It should be noted

that no crystal structures of 2-methylene-1,3-thiazolidin-4-one derivatives without a substituent at the N atom in position 3 of the thiazolidine ring were found.

5. Synthesis and crystallization

Thiazolidinones (I) and (II) were prepared from cyanoacetamides (see Fig. 7), by a previously described method (Obydenov *et al.*, 2017). Pyridine was added dropwise with stirring to cyanoacetamide (15 mmol) in a round-bottom flask until complete dissolution of the cyanoacetamide. 4-Dimethylaminopyridine (DMAP) (18 mg, 0.15 mmol) for (I), and mercaptoacetic acid (3.2 ml, 46 mmol) for (II), were added and the mixtures were refluxed for 12 h. They were then

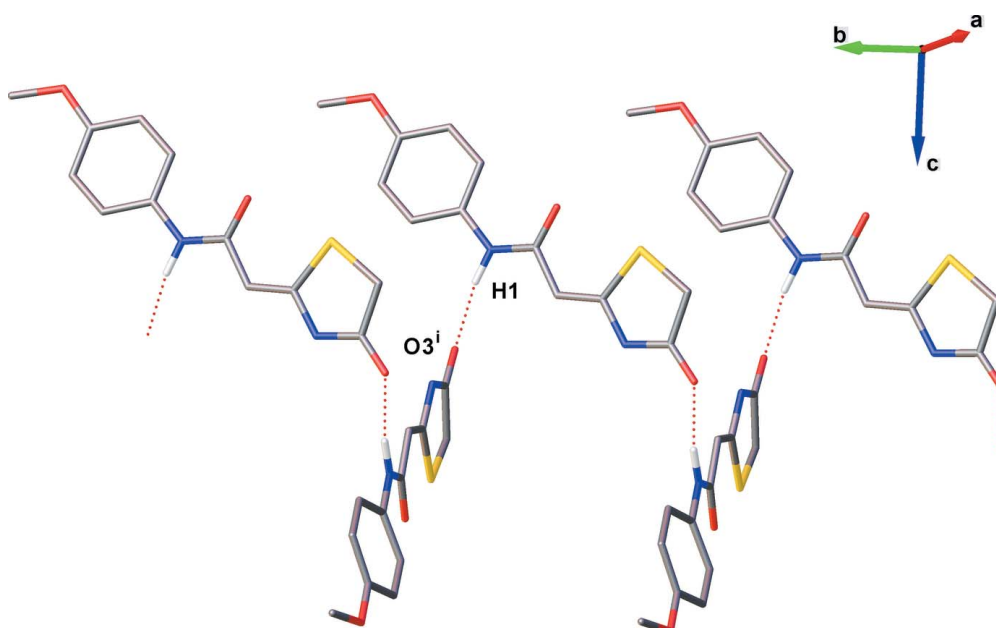


Figure 4
View of the N1—H1 \cdots O3ⁱ C(8) chain motif along the *b*-axis of compound (II). Dashed lines represent hydrogen bonds. For clarity, only the bridge H atoms are shown. [Symmetry code: (i) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.]

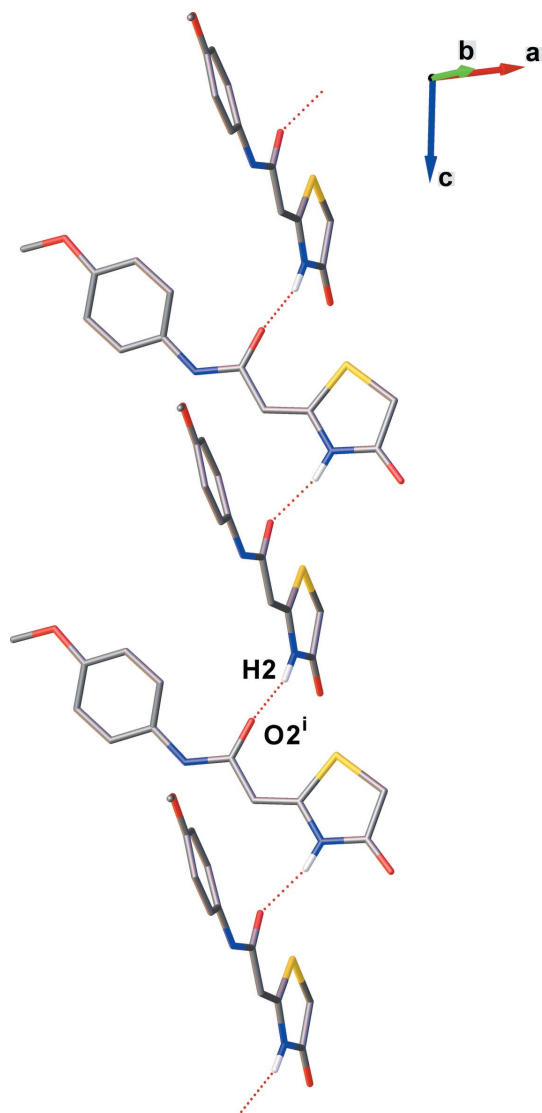


Figure 5

View of the $N2-H2 \cdots O2^i$ $C(6)$ chain motif along the c -axis of the compound (II). Dashed lines represent hydrogen bonds. For clarity, only the bridge H atoms are shown. [Symmetry code: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.]

cooled to room temperature and diluted with a 0.5 *N* HCl solution (5 ml). The precipitates formed of the 1,3-thiazolidinones, were filtered off. The crude products were additionally purified by refluxing a suspension of the thiazolidine in MeCN, followed by hot filtration. Colourless crystals of compounds (I) and (II) were obtained by slow evaporation of the respective compound in a solution of DMSO.

(Z)-2-[2-(Morpholin-4-yl)-2-oxoethylidene]-1,3-thiazolidin-4-one (I). Yield 1.54 g (45%), white powder, m.p. 503–505 K. 1H NMR spectrum, δ , p.p.m. (J , Hz): 3.42 (4H, *t*, 4.8 Hz, CH_2); 3.54 (2H, *s*, CH_2); 3.57 (4H, *t*, 4.8 Hz, CH_2); 5.83 (1H, *s*, CH); 11.25 (1H, *s*, NH).

(Z)-N-(4-Methoxyphenyl)-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamide (II). Yield 2.85 g (72%), white powder, m.p. 534–536 K. (Obydenov *et al.*, 2017). Elemental analysis for $C_{12}H_{12}N_2O_3S$; found, %: C 54.31; H 4.67; N 10.72; calculated, %: C 54.53; H 4.58; N 10.60.

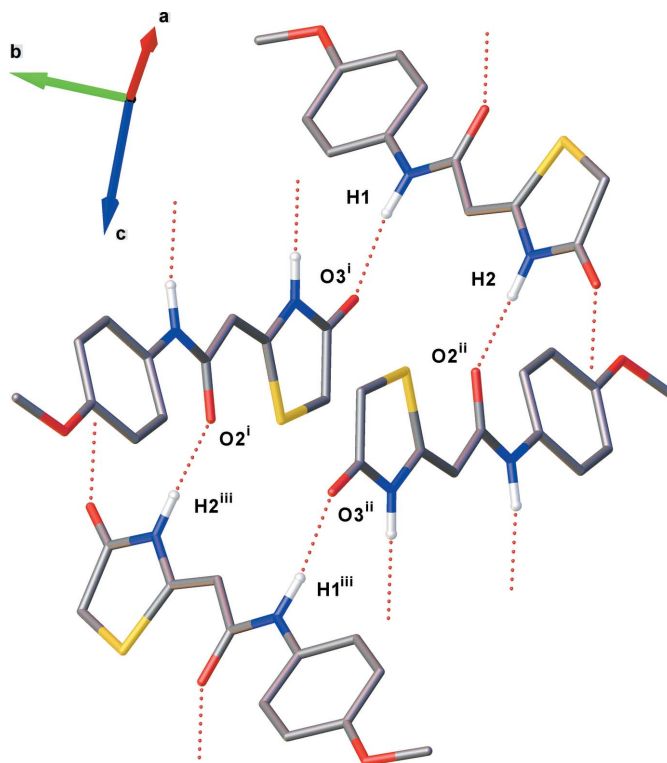


Figure 6

View of the tetrameric hydrogen-bonded aggregate which serves as the building block of the sheets. [Symmetry code: (i) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x, -y + 1, -z + 1$.]

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds, the hydrogen atoms were included in calculated positions and refined using the riding model: $C-H = 0.93-0.97$ Å with $U_{iso}(H) = 1.5U_{eq}(C\text{-methyl})$ and $1.2U_{eq}(C)$ for other C-bound H atoms. The NH H atoms were located in difference-Fourier maps and freely refined.

Funding information

This work was supported by the Russian Science Foundation (grant No. 16-16-04022).

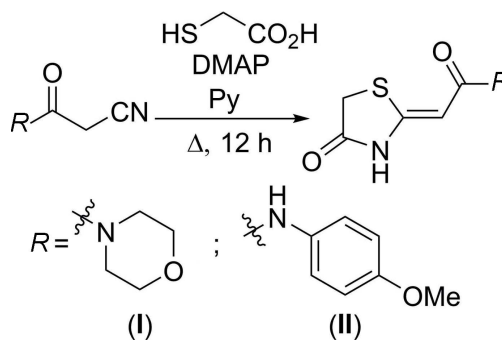


Figure 7

Reaction scheme for the title compounds.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C ₉ H ₁₂ N ₂ O ₃ S	C ₁₂ H ₁₂ N ₂ O ₃ S
<i>M</i> _r	228.27	264.30
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	295	295
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.9740 (4), 11.2175 (4), 9.3155 (4)	11.628 (11), 9.057 (6), 11.525 (12)
β (°)	100.389 (4)	101.13 (8)
<i>V</i> (Å ³)	1025.16 (7)	1190.8 (18)
<i>Z</i>	4	4
Radiation type	Mo <i>K</i> α	Cu <i>K</i> α
μ (mm ^{−1})	0.30	2.46
Crystal size (mm)	0.25 × 0.2 × 0.15	0.25 × 0.20 × 0.15
Data collection		
Diffractometer	Agilent Xcalibur Eos	Oxford Diffraction Xcalibur 3
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2013)	Multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2006)
<i>T</i> _{min} , <i>T</i> _{max}	0.924, 1.000	0.742, 1.000
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	5512, 2777, 2161	8436, 2040, 1398
<i>R</i> _{int}	0.017	0.053
(sin θ/λ) _{max} (Å ^{−1})	0.723	0.593
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.046, 0.154, 1.01	0.043, 0.105, 1.01
No. of reflections	2777	2040
No. of parameters	151	172
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ^{−3})	0.47, −0.24	0.22, −0.33

Computer programs: *CrysAlis PRO* (Agilent, 2013), *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2006), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008), *OLEX* (Dolomanov *et al.*, 2009) *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2011).

References

Agilent (2013). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, England.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

George, R. F. (2012). *Eur. J. Med. Chem.* **47**, 377–386.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.

Har, D. & Solensky, R. (2017). *Immunol. Allergy Clin. North Am.* **37**, 643–662.

Löscher, W. & Schmidt, D. (1994). *Epilepsy Res.* **17**, 95–134.

Obydenov, K. L., Galushchinskiy, A. N., Kosterina, M. F., Glukhareva, T. V. & Morzherin, Y. Y. (2017). *Chem. Heterocycl. Compd.* **53**, 622–625.

Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.

Rizos, C. V., Kei, A. & Elisaf, M. S. (2016). *Arch. Toxicol.* **90**, 1861–1881.

Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2017). E73, 1850-1854 [https://doi.org/10.1107/S2056989017016061]

Crystal structures of two (Z)-2-(4-oxo-1,3-thiazolidin-2-ylidene)acetamides

Aleksei Galushchinskiy, Pavel Slepukhin and Konstantin Obydenov

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2013) for (I); *CrysAlis CCD* (Oxford Diffraction, 2006) for (II). Cell refinement: *CrysAlis PRO* (Agilent, 2013) for (I); *CrysAlis RED* (Oxford Diffraction, 2006) for (II). Data reduction: *CrysAlis PRO* (Agilent, 2013) for (I); *CrysAlis RED* (Oxford Diffraction, 2006) for (II). For both structures, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008). Molecular graphics: *OLEX* (Dolomanov *et al.*, 2009) for (I); *SHELXTL* (Sheldrick, 2008) for (II). Software used to prepare material for publication: *PLATON* (Spek, 2009), *OLEX* (Dolomanov *et al.*, 2009) and *pubCIF* (Westrip, 2010) for (I); *SHELXTL* (Sheldrick, 2008) for (II).

(Z)-2-[2-(Morpholin-4-yl)-2-oxoethylidene]thiazolidin-4-one (I)

Crystal data

$C_9H_{12}N_2O_3S$

$M_r = 228.27$

Monoclinic, $P2_1/c$

$a = 9.9740$ (4) Å

$b = 11.2175$ (4) Å

$c = 9.3155$ (4) Å

$\beta = 100.389$ (4)°

$V = 1025.16$ (7) Å³

$Z = 4$

$F(000) = 480$

$D_x = 1.479$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å

Cell parameters from 1828 reflections

$\theta = 2.8$ – 30.1°

$\mu = 0.30$ mm⁻¹

$T = 295$ K

Prism, colourless

$0.25 \times 0.2 \times 0.15$ mm

Data collection

Agilent Xcalibur Eos
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 15.9555 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2013)

$T_{\min} = 0.924$, $T_{\max} = 1.000$

5512 measured reflections

2777 independent reflections

2161 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 30.9^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -14 \rightarrow 8$

$k = -12 \rightarrow 15$

$l = -8 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.154$

$S = 1.00$

2777 reflections

151 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.1P]$
where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F, with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.41099 (5)	0.19996 (4)	0.86944 (5)	0.04126 (18)
O1	0.60365 (15)	0.17462 (13)	0.70341 (18)	0.0530 (4)
O3	0.20314 (15)	0.45139 (14)	1.00948 (17)	0.0587 (4)
O2	1.01954 (17)	0.35357 (18)	0.6141 (3)	0.0846 (6)
N2	0.37439 (16)	0.42790 (15)	0.87892 (17)	0.0385 (3)
N1	0.74687 (16)	0.31439 (15)	0.6416 (2)	0.0475 (4)
C9	0.45545 (16)	0.34471 (15)	0.82632 (17)	0.0331 (3)
C11	0.27952 (17)	0.38781 (17)	0.95594 (19)	0.0385 (4)
C7	0.63595 (18)	0.28193 (17)	0.6972 (2)	0.0394 (4)
C8	0.55522 (18)	0.37426 (17)	0.7520 (2)	0.0393 (4)
H8	0.572 (2)	0.449 (2)	0.737 (2)	0.047*
C2	0.7968 (2)	0.43485 (19)	0.6280 (3)	0.0591 (6)
H2A	0.7513	0.4892	0.6844	0.071*
H2B	0.7764	0.4595	0.5266	0.071*
C10	0.2832 (2)	0.25413 (19)	0.9676 (2)	0.0441 (4)
C6	0.8258 (2)	0.2254 (2)	0.5779 (3)	0.0652 (7)
H6A	0.8106	0.2352	0.4728	0.078*
H6B	0.7957	0.1461	0.5990	0.078*
C3	0.9469 (3)	0.4400 (3)	0.6813 (4)	0.0813 (9)
H3A	0.9797	0.5188	0.6624	0.098*
H3B	0.9652	0.4275	0.7861	0.098*
C5	0.9720 (2)	0.2383 (2)	0.6379 (3)	0.0647 (6)
H5A	0.9876	0.2223	0.7419	0.078*
H5B	1.0231	0.1802	0.5925	0.078*
H10A	0.302 (3)	0.224 (3)	1.070 (3)	0.075 (8)*
H10B	0.195 (4)	0.232 (3)	0.933 (3)	0.089 (10)*
H2	0.377 (3)	0.498 (3)	0.869 (2)	0.060 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0494 (3)	0.0229 (3)	0.0547 (3)	-0.00327 (18)	0.0178 (2)	-0.00108 (17)

O1	0.0573 (8)	0.0254 (7)	0.0828 (10)	−0.0052 (6)	0.0301 (7)	−0.0098 (6)
O3	0.0605 (9)	0.0382 (8)	0.0881 (10)	0.0025 (7)	0.0422 (8)	0.0000 (7)
O2	0.0595 (10)	0.0483 (11)	0.1621 (18)	−0.0058 (8)	0.0631 (11)	−0.0175 (12)
N2	0.0387 (7)	0.0234 (8)	0.0568 (9)	−0.0013 (6)	0.0173 (6)	−0.0008 (6)
N1	0.0405 (8)	0.0293 (9)	0.0774 (11)	−0.0024 (6)	0.0237 (7)	−0.0110 (7)
C9	0.0346 (8)	0.0226 (8)	0.0419 (8)	−0.0024 (6)	0.0067 (6)	−0.0032 (6)
C11	0.0377 (8)	0.0302 (9)	0.0491 (9)	−0.0032 (7)	0.0119 (7)	−0.0002 (7)
C7	0.0377 (8)	0.0290 (9)	0.0531 (10)	−0.0011 (7)	0.0128 (7)	−0.0052 (7)
C8	0.0392 (8)	0.0221 (8)	0.0597 (10)	−0.0015 (7)	0.0171 (7)	−0.0013 (7)
C2	0.0575 (11)	0.0298 (10)	0.1001 (16)	0.0062 (9)	0.0408 (11)	0.0098 (10)
C10	0.0476 (10)	0.0321 (10)	0.0562 (11)	−0.0026 (8)	0.0195 (8)	0.0039 (8)
C6	0.0599 (13)	0.0455 (13)	0.0992 (18)	−0.0064 (11)	0.0386 (12)	−0.0288 (12)
C3	0.0588 (13)	0.0460 (15)	0.151 (3)	−0.0153 (11)	0.0519 (15)	−0.0309 (16)
C5	0.0581 (13)	0.0446 (13)	0.1004 (18)	0.0148 (11)	0.0380 (12)	0.0041 (12)

Geometric parameters (Å, °)

S1—C9	1.7490 (17)	C7—C8	1.459 (2)
S1—C10	1.803 (2)	C8—H8	0.87 (3)
O1—C7	1.250 (2)	C2—H2A	0.9700
O3—C11	1.214 (2)	C2—H2B	0.9700
O2—C3	1.422 (3)	C2—C3	1.490 (3)
O2—C5	1.408 (3)	C10—H10A	0.99 (3)
N2—C9	1.381 (2)	C10—H10B	0.91 (3)
N2—C11	1.363 (2)	C6—H6A	0.9700
N2—H2	0.79 (3)	C6—H6B	0.9700
N1—C7	1.353 (2)	C6—C5	1.472 (3)
N1—C2	1.454 (3)	C3—H3A	0.9700
N1—C6	1.462 (3)	C3—H3B	0.9700
C9—C8	1.352 (2)	C5—H5A	0.9700
C11—C10	1.503 (3)	C5—H5B	0.9700
C9—S1—C10	92.02 (8)	C3—C2—H2B	109.6
C5—O2—C3	110.09 (19)	S1—C10—H10A	109.8 (18)
C9—N2—H2	127 (2)	S1—C10—H10B	117 (2)
C11—N2—C9	118.05 (16)	C11—C10—S1	108.05 (13)
C11—N2—H2	115 (2)	C11—C10—H10A	114.0 (17)
C7—N1—C2	126.90 (17)	C11—C10—H10B	104 (2)
C7—N1—C6	120.57 (17)	H10A—C10—H10B	105 (3)
C2—N1—C6	112.42 (17)	N1—C6—H6A	109.6
N2—C9—S1	110.91 (12)	N1—C6—H6B	109.6
C8—C9—S1	125.85 (14)	N1—C6—C5	110.34 (19)
C8—C9—N2	123.23 (16)	H6A—C6—H6B	108.1
O3—C11—N2	124.68 (18)	C5—C6—H6A	109.6
O3—C11—C10	124.40 (16)	C5—C6—H6B	109.6
N2—C11—C10	110.91 (16)	O2—C3—C2	112.8 (2)
O1—C7—N1	120.81 (17)	O2—C3—H3A	109.0
O1—C7—C8	120.27 (17)	O2—C3—H3B	109.0

N1—C7—C8	118.92 (17)	C2—C3—H3A	109.0
C9—C8—C7	120.57 (17)	C2—C3—H3B	109.0
C9—C8—H8	119.9 (15)	H3A—C3—H3B	107.8
C7—C8—H8	119.5 (16)	O2—C5—C6	111.7 (2)
N1—C2—H2A	109.6	O2—C5—H5A	109.3
N1—C2—H2B	109.6	O2—C5—H5B	109.3
N1—C2—C3	110.2 (2)	C6—C5—H5A	109.3
H2A—C2—H2B	108.1	C6—C5—H5B	109.3
C3—C2—H2A	109.6	H5A—C5—H5B	107.9
S1—C9—C8—C7	1.1 (3)	C7—N1—C2—C3	133.5 (2)
O1—C7—C8—C9	8.3 (3)	C7—N1—C6—C5	−130.8 (2)
O3—C11—C10—S1	179.35 (16)	C2—N1—C7—O1	−179.8 (2)
N2—C9—C8—C7	−179.01 (15)	C2—N1—C7—C8	−0.6 (3)
N2—C11—C10—S1	−1.38 (19)	C2—N1—C6—C5	52.9 (3)
N1—C7—C8—C9	−170.79 (18)	C10—S1—C9—N2	−2.10 (14)
N1—C2—C3—O2	53.1 (3)	C10—S1—C9—C8	177.80 (17)
N1—C6—C5—O2	−57.3 (3)	C6—N1—C7—O1	4.4 (3)
C9—S1—C10—C11	1.95 (14)	C6—N1—C7—C8	−176.4 (2)
C9—N2—C11—O3	179.09 (17)	C6—N1—C2—C3	−50.5 (3)
C9—N2—C11—C10	−0.2 (2)	C3—O2—C5—C6	59.6 (3)
C11—N2—C9—S1	1.7 (2)	C5—O2—C3—C2	−57.7 (3)
C11—N2—C9—C8	−178.18 (16)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O1 ⁱ	0.79 (3)	2.11 (3)	2.891 (2)	167 (2)
C2—H2A \cdots S1 ⁱ	0.97	2.86	3.627 (2)	137
C5—H5B \cdots O3 ⁱⁱ	0.97	2.55	3.503 (3)	167
C6—H6B \cdots O3 ⁱⁱⁱ	0.97	2.41	3.179 (3)	136

Symmetry codes: (i) $-x+1, y+1/2, -z+3/2$; (ii) $x+1, -y+1/2, z-1/2$; (iii) $-x+1, y-1/2, -z+3/2$.

(Z)-N-(4-Methoxyphenyl)-2-(4-oxothiazolidin-2-ylidene)acetamide (II)

Crystal data

C₁₂H₁₂N₂O₃S

M_r = 264.30

Monoclinic, *P*2₁/*c*

Hall symbol: -P 2ybc

a = 11.628 (11) Å

b = 9.057 (6) Å

c = 11.525 (12) Å

β = 101.13 (8)°

V = 1190.8 (18) Å³

Z = 4

F(000) = 552

D_x = 1.474 Mg m^{−3}

Cu *K*α radiation, λ = 1.54184 Å

Cell parameters from 3173 reflections

θ = 3.8–65.3°

μ = 2.46 mm^{−1}

T = 295 K

Prism, colourless

0.25 × 0.20 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur 3
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 ω scans
Absorption correction: multi-scan
(CrysAlis RED; Oxford Diffraction, 2006)
 $T_{\min} = 0.742$, $T_{\max} = 1.000$

8436 measured reflections
2040 independent reflections
1398 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$
 $\theta_{\max} = 66.2^\circ$, $\theta_{\min} = 3.9^\circ$
 $h = -12 \rightarrow 13$
 $k = -9 \rightarrow 10$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.105$
 $S = 1.01$
2040 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.060P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.12936 (5)	0.11898 (8)	−0.00103 (5)	0.0502 (2)
N1	−0.17601 (18)	0.4274 (3)	−0.04195 (17)	0.0492 (5)
O1	−0.46385 (19)	0.6658 (2)	−0.43527 (18)	0.0749 (6)
C1	−0.2477 (2)	0.4870 (3)	−0.1432 (2)	0.0455 (6)
O2	−0.04377 (14)	0.2965 (2)	−0.12198 (14)	0.0547 (5)
N2	0.10328 (18)	0.1413 (3)	0.21526 (18)	0.0512 (6)
C2	−0.3184 (2)	0.6044 (3)	−0.1284 (2)	0.0509 (6)
H2A	−0.3151	0.6431	−0.0531	0.061*
O3	0.24270 (17)	−0.0064 (2)	0.31929 (16)	0.0660 (6)
C3	−0.3939 (2)	0.6656 (3)	−0.2224 (2)	0.0575 (7)
H3A	−0.4420	0.7441	−0.2108	0.069*
C4	−0.3975 (2)	0.6099 (3)	−0.3341 (2)	0.0530 (6)
C5	−0.3311 (2)	0.4883 (3)	−0.3482 (2)	0.0552 (7)
H5A	−0.3376	0.4468	−0.4230	0.066*
C6	−0.2555 (2)	0.4267 (3)	−0.2548 (2)	0.0510 (6)

H6A	−0.2101	0.3455	−0.2663	0.061*
C7	−0.0826 (2)	0.3377 (3)	−0.0346 (2)	0.0450 (6)
C8	−0.0324 (2)	0.2893 (3)	0.0828 (2)	0.0476 (6)
H8A	−0.0631	0.3248	0.1462	0.057*
C9	0.0570 (2)	0.1951 (3)	0.10347 (19)	0.0443 (6)
C10	0.2276 (2)	0.0170 (3)	0.1101 (2)	0.0528 (7)
H10A	0.2212	−0.0879	0.0930	0.063*
H10B	0.3079	0.0473	0.1117	0.063*
C11	0.1943 (2)	0.0481 (3)	0.2273 (2)	0.0512 (6)
C12	−0.5271 (3)	0.7961 (4)	−0.4254 (3)	0.0815 (10)
H12A	−0.5700	0.8240	−0.5019	0.122*
H12B	−0.4736	0.8737	−0.3945	0.122*
H12C	−0.5807	0.7796	−0.3730	0.122*
H1	−0.198 (2)	0.461 (3)	0.029 (2)	0.058 (7)*
H2	0.068 (3)	0.163 (3)	0.279 (2)	0.067 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0518 (3)	0.0662 (4)	0.0356 (3)	−0.0033 (3)	0.0159 (2)	−0.0037 (3)
N1	0.0515 (11)	0.0638 (14)	0.0335 (11)	0.0009 (10)	0.0108 (9)	−0.0024 (10)
O1	0.0742 (13)	0.0805 (15)	0.0637 (13)	0.0219 (11)	−0.0025 (10)	0.0061 (11)
C1	0.0425 (12)	0.0557 (15)	0.0392 (13)	−0.0076 (11)	0.0106 (10)	−0.0008 (11)
O2	0.0519 (9)	0.0796 (13)	0.0353 (9)	0.0040 (9)	0.0151 (8)	0.0008 (9)
N2	0.0537 (12)	0.0693 (15)	0.0338 (11)	0.0037 (11)	0.0167 (9)	0.0020 (10)
C2	0.0518 (13)	0.0555 (16)	0.0470 (14)	−0.0058 (13)	0.0136 (11)	−0.0108 (12)
O3	0.0672 (12)	0.0876 (15)	0.0454 (12)	0.0110 (10)	0.0165 (9)	0.0173 (10)
C3	0.0524 (14)	0.0550 (16)	0.0645 (19)	0.0025 (12)	0.0097 (13)	−0.0084 (13)
C4	0.0478 (13)	0.0605 (16)	0.0496 (15)	−0.0001 (13)	0.0068 (11)	0.0057 (13)
C5	0.0561 (14)	0.0723 (19)	0.0381 (14)	0.0075 (14)	0.0113 (11)	−0.0013 (12)
C6	0.0526 (13)	0.0635 (17)	0.0384 (13)	0.0071 (13)	0.0126 (11)	−0.0016 (12)
C7	0.0431 (12)	0.0559 (15)	0.0384 (14)	−0.0087 (11)	0.0138 (10)	−0.0031 (11)
C8	0.0485 (13)	0.0624 (17)	0.0342 (13)	−0.0058 (12)	0.0141 (10)	−0.0036 (11)
C9	0.0471 (12)	0.0549 (16)	0.0328 (13)	−0.0093 (12)	0.0126 (10)	0.0006 (11)
C10	0.0570 (14)	0.0615 (17)	0.0430 (15)	−0.0022 (13)	0.0179 (11)	−0.0034 (12)
C11	0.0521 (13)	0.0652 (17)	0.0383 (14)	−0.0058 (13)	0.0138 (11)	0.0053 (13)
C12	0.0685 (19)	0.079 (2)	0.094 (3)	0.0220 (18)	0.0093 (17)	0.0164 (19)

Geometric parameters (Å, °)

S1—C9	1.739 (3)	C3—C4	1.375 (4)
S1—C10	1.798 (3)	C3—H3A	0.9300
N1—C7	1.346 (3)	C4—C5	1.372 (4)
N1—C1	1.405 (3)	C5—C6	1.370 (4)
N1—H1	0.96 (3)	C5—H5A	0.9300
O1—C4	1.365 (3)	C6—H6A	0.9300
O1—C12	1.407 (4)	C7—C8	1.435 (4)
C1—C2	1.375 (4)	C8—C9	1.330 (4)

C1—C6	1.384 (3)	C8—H8A	0.9300
O2—C7	1.238 (3)	C10—C11	1.501 (4)
N2—C11	1.339 (4)	C10—H10A	0.9700
N2—C9	1.385 (3)	C10—H10B	0.9700
N2—H2	0.92 (3)	C12—H12A	0.9600
C2—C3	1.373 (4)	C12—H12B	0.9600
C2—H2A	0.9300	C12—H12C	0.9600
O3—C11	1.206 (3)		
C9—S1—C10	92.05 (13)	C1—C6—H6A	120.4
C7—N1—C1	128.8 (2)	O2—C7—N1	123.1 (2)
C7—N1—H1	119.0 (16)	O2—C7—C8	122.0 (2)
C1—N1—H1	112.1 (16)	N1—C7—C8	114.9 (2)
C4—O1—C12	117.4 (2)	C9—C8—C7	121.7 (2)
C2—C1—C6	119.1 (2)	C9—C8—H8A	119.1
C2—C1—N1	117.9 (2)	C7—C8—H8A	119.1
C6—C1—N1	122.8 (2)	C8—C9—N2	122.8 (2)
C11—N2—C9	118.4 (2)	C8—C9—S1	126.60 (19)
C11—N2—H2	121.0 (17)	N2—C9—S1	110.58 (19)
C9—N2—H2	120.5 (18)	C11—C10—S1	107.8 (2)
C3—C2—C1	121.4 (2)	C11—C10—H10A	110.1
C3—C2—H2A	119.3	S1—C10—H10A	110.1
C1—C2—H2A	119.3	C11—C10—H10B	110.1
C2—C3—C4	119.3 (3)	S1—C10—H10B	110.1
C2—C3—H3A	120.3	H10A—C10—H10B	108.5
C4—C3—H3A	120.3	O3—C11—N2	125.1 (2)
O1—C4—C5	115.7 (2)	O3—C11—C10	123.8 (3)
O1—C4—C3	125.0 (3)	N2—C11—C10	111.1 (2)
C5—C4—C3	119.3 (2)	O1—C12—H12A	109.5
C6—C5—C4	121.6 (2)	O1—C12—H12B	109.5
C6—C5—H5A	119.2	H12A—C12—H12B	109.5
C4—C5—H5A	119.2	O1—C12—H12C	109.5
C5—C6—C1	119.1 (3)	H12A—C12—H12C	109.5
C5—C6—H6A	120.4	H12B—C12—H12C	109.5
C7—N1—C1—C2	−163.6 (2)	C1—N1—C7—C8	−176.5 (2)
C7—N1—C1—C6	20.8 (4)	O2—C7—C8—C9	−1.5 (4)
C6—C1—C2—C3	−2.0 (4)	N1—C7—C8—C9	176.9 (2)
N1—C1—C2—C3	−177.8 (2)	C7—C8—C9—N2	−176.8 (2)
C1—C2—C3—C4	−1.0 (4)	C7—C8—C9—S1	1.6 (4)
C12—O1—C4—C5	−175.9 (3)	C11—N2—C9—C8	−179.2 (2)
C12—O1—C4—C3	4.5 (4)	C11—N2—C9—S1	2.2 (3)
C2—C3—C4—O1	−176.4 (2)	C10—S1—C9—C8	179.6 (2)
C2—C3—C4—C5	4.1 (4)	C10—S1—C9—N2	−1.85 (19)
O1—C4—C5—C6	176.1 (2)	C9—S1—C10—C11	1.19 (19)
C3—C4—C5—C6	−4.2 (4)	C9—N2—C11—O3	179.9 (2)
C4—C5—C6—C1	1.2 (4)	C9—N2—C11—C10	−1.2 (3)
C2—C1—C6—C5	1.9 (4)	S1—C10—C11—O3	178.6 (2)

N1—C1—C6—C5	177.4 (2)	S1—C10—C11—N2	−0.2 (3)
C1—N1—C7—O2	1.9 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O3 ⁱ	0.95 (2)	1.94 (2)	2.883 (4)	170 (2)
N2—H2 \cdots O2 ⁱⁱ	0.93 (3)	1.92 (3)	2.828 (4)	164 (3)

Symmetry codes: (i) $-x, y+1/2, -z+1/2$; (ii) $x, -y+1/2, z+1/2$.